

California Environmental Protection Agency

---



**SOP MLD 029**

**STANDARD OPERATING PROCEDURE FOR  
ANALYZING THE MASS OF  
DICHOTOMOUS PM10 FILTERS**

Northern Laboratory Branch  
Monitoring and Laboratory Division

First Approved Date of SOP: October 1, 1991  
Approval Date of Last SOP Amendment: January 2, 2002  
Revision Number: 2.0

DISCLAIMER: Mention of any trade name or commercial product in this Standard Operating Procedure does not constitute endorsement or recommendation of this product by the Air Resources Board. Specific brand names and instrument descriptions listed in the Standard Operating Procedure are for equipment used by the ARB laboratory.

## **Table of Contents**

**1**

<b>1.0</b>	<b>SCOPE .....</b>	<b>1</b>
<b>2.0</b>	<b>SUMMARY OF METHOD.....</b>	<b>1</b>
<b>3.0</b>	<b>INTERFERENCES .....</b>	<b>1</b>
<b>4.0</b>	<b>APPARATUS AND MATERIALS.....</b>	<b>2</b>
<b>5.0</b>	<b>BALANCE CALIBRATION PROCEDURE.....</b>	<b>2</b>
<b>6.0</b>	<b>FILTER INSPECTION AND EQUILIBRATION--PRE-WEIGHTS.....</b>	<b>3</b>
<b>7.0</b>	<b>FILTER PRE-WEIGHT .....</b>	<b>3</b>
<b>8.0</b>	<b>FILTER LOGIN AFTER FIELD SAMPLING.....</b>	<b>5</b>
<b>9.0</b>	<b>FILTER POST-WEIGHING.....</b>	<b>5</b>
<b>APPENDIX A:</b>	<b>DICHOTOMOUS PM10 INVALIDATION CRITERIA .....</b>	<b>7</b>

## **SOP MLD 029**

### **STANDARD OPERATING PROCEDURE FOR ANALYZING THE MASS OF DICHOTOMOUS PM<sub>10</sub> FILTERS**

#### **1.0 SCOPE**

This document describes the methodology used by MLD staff to analyze the mass of Dichotomous PM<sub>10</sub> filter samples.

#### **2.0 SUMMARY OF METHOD**

Teflon filters (37-mm diameter) are weighed on an electronic microbalance before and after field sampling. A sample consists of a filter set: a coarse filter onto which 2.5 to 10  $\mu\text{m}$  particles are deposited, and a fine filter that collects particles less than 2.5  $\mu\text{m}$ . Filter weights are recorded directly employing a microbalance-computer interface that calculates the mass of particles per cubic meter of air ( $\mu\text{g}/\text{m}^3$ ).

#### **3.0 INTERFERENCES**

- 3.1 The potential effect of body moisture or oils contacting the filters is minimized by using non-serrated forceps to handle the filters at all times. This measure also moderates interference due to static electricity.
- 3.2 Teflon filters accumulate a surface electrical charge that may affect filter weight. Treating filters on a "Static Master" prior to weighing controls static electricity.
- 3.2 Moisture content will affect filter weight. Filters must be equilibrated for a minimum of 24 hours in a controlled environment prior to pre- and post-weighing. During this equilibration period, the relative humidity (RH) must remain between 20 and 45%  $\pm$  5%RH and the temperature at between 15 and 30  $^{\circ}\text{C} \pm 3.0^{\circ}\text{C}$ . The goal during the equilibration period will be to maintain a relative humidity of 35%  $\pm$  5%RH and a temperature of 23 $^{\circ}\text{C} \pm 3.0^{\circ}\text{C}$ . If the relative humidity and temperature fall outside of these ranges at any time during the equilibration period, the equilibration must be started over.

The temperature and relative humidity are recorded on a Dickson Humidity/Temperature weekly chart recorder. The humidity / temperature recorder is calibrated every three months using a NIST traceable humidity/temperature monitor. The humidity (RH) and temperature ( $^{\circ}\text{C}$ ) must be within  $\pm 2$  of the NIST standard value.

- 3.4 Airborne particles can interfere with the weight of the filters. Equilibrating filters on racks in a vented cabinet prevents such contamination.

#### **4.0 APPARATUS AND MATERIALS**

- 4.1 Sartorius M5P microbalance with a minimum resolution of 0.001 mg and a precision of  $\pm 0.005$  mg. A balance pan should also be included.
- 4.2 Troemner Class 1.1 Calibration Weights or equivalent (specifically a 100-mg weight).
- 4.3 Computer terminal interfaced to LIMS (Laboratory Information Management System)
- 4.4 Nuclear Products "Static Master" anti-static devices (4 ea.).
- 4.5 Forceps.
- 4.6 Filter: Teflon 37 mm PTFE membrane (Gelman Science Teflon #R2PJ037)
- 4.7 Filter Holder Support Rings.
- 4.8 Petri Dishes (Falcon #1006) 50 x 9 mm.
- 4.9 Filter Equilibration Racks.
- 4.10 Dickson Model THDX Humidity/Temperature Recorder.
- 4.11 Digital Timer/Stopwatch (VWR).

#### **5.0 BALANCE CALIBRATION PROCEDURE**

- 5.1 Prior to weighing a filter, the balance must be calibrated. First check the balance level (re-level if necessary). The balance display will read "standby." Press the on/off key to activate the balance. The balance performs an internal circuitry check, which is complete, when "CH2" appears in the Liquid Crystal Display (LCD). The LCD then displays a "L" which indicates the load weights should be removed (i.e., load weights are used only for weighing objects in excess of 750 or 1500 mg). Press the small "T" to remove the load weights.
- 5.2 Internal Calibration: Once the stabilization bubble (hereafter referred to as the "bubble") appears above "mg," press the "CAL" key. Once the "bubble" re-appears, press the "CAL" key again. The balance then displays "C." Two different calibrations are performed. The second calibration is indicated by

"CC." Should the display read "CE," an error occurred and you must recalibrate. Once the internal calibration is complete, the display should read "0.000" with a bubble above the "mg." The balance is now ready for external calibration.

- 5.3 External Calibration: Open the chamber door and with plastic forceps, place the 100-mg weight into the chamber. Close the door and record the temperature and relative humidity of the room in the "QC M5P Balance Calibration Notebook". Once the bubble appears, start timing with the stopwatch for 30 seconds. Remove the 100-mg weight from balance and record the mass of the 100-mg weight into the calibration notebook. The weight must be  $100 \pm 0.005$  mg. If the weight is within the control limit go to section 5.4. If the weight exceeds the control limit, the balance must be re-calibrated using a primary (certified) 100-mg weight (repeat section 5.2 to 5.3). If the re-calibration weight still exceeds the control limit, call service to check the balance.
- 5.4 Enter the information recorded in the "QC M5P Balance Calibration Notebook" into LIMS. Open Procomm4 and select SQL\*LIMS from the left drop-down box at the telnet prompt. Enter a username and password to log onto LIMS. Select LOG, then ENTER, and BY SAMPLE PLAN to begin entering data. Enter the appropriate balance name, then page down to enter the information from the notebook. Record the LIMS number found at the top right of the screen in the calibration notebook.

## **6.0 FILTER INSPECTION AND EQUILIBRATION--PRE-WEIGHTS**

The filters are equilibrated for a minimum of 24 hours in a closed but vented cabinet. Using forceps, all filters are visually inspected for defects or irregularities that may interfere with sampling or mass analyses. If the balance room conditions are out of range for either the temperature or relative humidity standard (as described in Section 3.3) no weighing can be done and filters must be re-equilibrated for an additional 24 hours.

## **7.0 FILTER PRE-WEIGHT**

- 7.1 To pre-weigh filters, the balance must be calibrated and the filters must be equilibrated (see Sections 5 and 6).
- 7.2 Prepare a set of 24-Hour Report Forms equal to the number of dichot filter pairs that will be sent out. Place corresponding barcodes on the filter sheets and on the designated coarse and fine Petri dishes. Make sure that the barcodes match during the weighing session.
- 7.3 Open Procomm4 and select sql\*lims2 from the right drop-down window.

Select yes to initiate the weighing session. Select the "PRE" option, then the dichot program. Scan or enter the barcode from the 24-Hour Report Form. The computer will prompt you to weigh the coarse filter first.

- 7.4 Place a filter set on the Static Master for approximately 30 seconds. Remember to handle the filters only with forceps and hold only the filter's outer ring.
- 7.5 Place the coarse filter in the balance chamber and close the door. While waiting for the bubble to appear, initial and date the 24-Hour Sample Report Form. Begin timing for 30 seconds once the bubble appears. At 30 seconds, press the Print key to transmit the weight to the computer. Record the weight on the 24-Hour Sample Report Form. Place the filter in yellow support rings and enclose it in the coarse Petri dish. For proper sampling, the filter surface with the support "lip" should be placed right side up.
- 7.6 The computer then prompts you to weigh the fine filter. Again, 30 seconds after the bubble appears, press the Print key to transmit the fine filter weight. Record the weight on the 24-Hour Sample Report Form. Place the filter in white support rings and enclose it in the fine Petri dish. Again, for proper sampling, the filter surface with the support "lip" should be placed right side up.
- 7.7 After each filter set is weighed, check the balance zero. If the balance does not re-zero after the bubble appears, press TARE.
- 7.8 Repeat the above procedure for an additional nine filter sets. After weighing 10 filters sets, a duplicate filter pair must be weighed as a Quality Control Check (i.e., duplicates must equal 10% of the number of total weights recorded). Duplicate weights must fall within  $\pm 20 \mu\text{g}$  of the original value (according to EPA protocol). Reweigh the last filter pair. Proceed as usual and record the duplicate weight on the 24-Hour Sample Report Form in the spaces provided.
- 7.9 After pre-weighing is complete, a text file is generated that must be transferred into LIMS. This is accomplished through a file transfer protocol (FTP). Note the filename assigned to the file, then enter the following at the MS\_DOS prompt:

```
cd c:\temp\  
ftp  
open 146.114.112.200  
(enter username) balance  
(enter password) balance  
put (filename)
```

quit  
exit

Once the file has been transferred into LIMS, a confirmation report will print. Generate a pre-weight report by selecting and printing the **dipre.rep** file. Enter the filename printed at the top of the confirmation report. Once the pre-weight report is generated, place it in the Dichot pre-weight summary binder.

## 8.0 FILTER LOGIN AFTER FIELD SAMPLING

- 8.1 Exposed filter samples with a completed 24-Hour Sample Report Form are received via US Mail. Inspect the filter set for possible defects or irregular sample loading [Note: Use Appendix A to assist you]. Remove the samples from their holders and place them upright into the Petri dish. Verify the information on the 24-Hour Sample Report Form and record the date received.
- 8.2 Open Procomm4 and select SQL\*LIMS from the left drop-down box at the telnet prompt. Enter a username and password to log onto LIMS. Select LOG, then ENTER, and BY SAMPLE PLAN to begin entering data. Enter the appropriate dichot site name and scan or enter the barcode on the 24-Hour Sample Report Form. Using the data on the 24-Hour Sample Report Form, type in the appropriate response to each prompt.
- 8.3 Equilibrate exposed filters for 24 hours in open Petri dishes in a closed, but vented cabinet.

## 9.0 FILTER POST-WEIGHING

- 9.1 After the filters have equilibrated for at least 24 hours under the required conditions of temperature and humidity, generate a mass worklist from the LIMS directory. Select and print the file **diwrkms.rep**.
- 9.2 Check the worklist for the appropriate number of duplicates (10%).
- 9.3 To post-weigh samples, the microbalance is calibrated as described in Section 5.0.
- 9.4 Open Procomm4 and select sql\*lims2 from the right drop-down window. Select yes to initiate the weighing session. Select the "POST" option, then the dichot program. Scan or enter the barcode from the 24-Hour Sample Report Form that corresponds to the samples listed on the mass worklist. The computer will prompt you to weigh the coarse filter first.

- 9.5 As with pre-weighing, place the first sample set on the Static Master for approximately 30 seconds. Remember that filters are only handled with forceps. Place the coarse sample in the balance chamber. When the bubble appears, begin timing for 30 seconds. At 30 seconds, transmit the post-weight to the computer. Record this weight on the 24-Hour Sample Report Form.
- 9.6 Follow the same procedure for weighing the fine sample. After each sample set is weighed, recheck the balance zero. Tare as necessary.
- 9.7 The worklist will generate duplicate samples for a Quality Control check. Reweigh the duplicate sample after placing the filters on the Static Master for 30 seconds. Record this information on the 24-Hour Sample Report Form in the spaces provided.
- 9.8 After post-weighing is complete, a text file is generated that must be transferred into LIMS. This is accomplished through a file transfer protocol (FTP). Note the filename assigned to the file, then enter the following at the MS\_DOS prompt:

```
cd c:\temp\  
ftp  
open 146.114.112.200  
(enter username) balance  
(enter password) balance  
put (filename)  
quit  
exit
```

Once the file has been transferred into LIMS, a confirmation report will print. Generate a post-weight report by selecting and printing the file **dipost.rep**. Enter the filename printed at the top of the confirmation report. Once the post-weight report is generated, place it in the Dichot post-weight summary binder.

- 9.9 Bind post-weighed samples in a group of 10 (5 pair) with rubber bands and place in a small box. The x-ray fluorescence analyst will inspect the samples under a microscope to determine if the sampler deposited metal particles on the filters. All samples will be returned to the balance room after analysis for archiving. The sample that is contaminated and will affect the x-ray fluorescence analysis will be invalid in the LIMS by balance room analyst.
- 9.10 Save post-weighed samples in sectioned archive boxes for x-ray fluorescence analysis. Samples from two months are placed in an archive box alphabetically by site with the fine fraction on top. Place the LIMS report **distatus.rep** and the



24-Hour Sample Report Forms for the month arranged alphabetically by site in the box. Each site and corresponding report forms are grouped in chronological order by sample date. Store in a cool dry place until retrieved by the x-ray fluorescence analyst.

## **APPENDIX A: DICHOTOMOUS PM10 INVALIDATION CRITERIA**

### **1.0 SCOPE**

Quality control invalidation criteria for PM10 dichotomous (Dichot) filter samples are listed below. All samples taken in the field will be checked using these criteria. If a sample is found to be invalid, the sample will be sent to the laboratory with the 24-Hour Sample Report Form. If only one of the pair of dichot filters is invalidated, both the coarse filter and the fine filter should be considered invalid.

Because these data are used to determine the fractionated size of the components of PM10 rather than to determine attainment status, a make-up run is not necessary for invalidated dichotomous sampler runs. However, every effort should be made to ensure proper sampler operation so the next scheduled sample run produces valid data. If consecutive runs do not produce valid data, the Air Quality Surveillance (AQS) section manager must be notified immediately so corrective action can be taken.

### **2.0 DICHOTOMOUS PM10 FILTER INVALIDATION CRITERIA**

#### **2.1 Filter Contamination**

Filters dropped on counters or the floor, or found with visible scratches either before or after sampling should be invalidated.

#### **2.2 Damaged or Torn Filters**

Regardless of whether the damage occurs before or after sampling, torn filters or filters with pinholes should be invalidated. If only one of the pair of dichot filters is damaged or torn, both the coarse and fine filter should be considered invalid.

#### **2.3 Dichotomous Sampler Flowrate**

The average flowrate must be maintained within  $\pm 10$  percent of 16.7 liters per minute at actual conditions to ensure proper particle fractionation of the sample. This would allow flowrates from 15.0 to 18.4 liters per minute.

Therefore, if the total sample flowrate, as observed by the rotometer reading before and after the run, varies from 16.7 liters per minute by greater than 10% due to equipment failure or operator error, the sample shall be invalidated because the 10 micrometer and dichotomous cutpoints cannot be maintained.

If these flowrate limits are exceeded, due to instrument malfunction, the station operator will correct the malfunction to ensure proper sampler operation on the next scheduled sample run day.

If the total sample flowrate, as observed by the rotometer reading, decreases more than 10% from 16.7 liters per minute because of heavy particulate loading on the filter, a post sampling check of the vacuum gauges will indicate an increased vacuum and an inspection of the filter will reveal heavy particulate loading. These filters should **not** be invalidated because they may indicate an episode of a high concentration of airborne particulate matter. These samples will be analyzed and the data flagged.

## 2.4 Elapsed Time

All samplers must turn ON and OFF within 30 minutes of midnight. (23:30 – 00:30)

All samplers must operate for at least 23 but no more than 25 hours (1380 to 1500 minutes).

Samples running outside of these limits should be sent to the laboratory. These samples can still provide useful information and will be analyzed with the data flagged, even though they do not meet sampling criteria.